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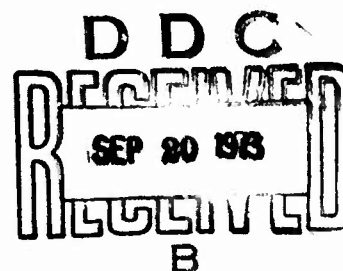
AFML-TR-73-101

**GRAPHITE FIBER AND BORON NITRIDE FIBER  
FILLED POTTING COMPOUNDS**

*R. J. DAUKSYS*

TECHNICAL REPORT AFML-TR-73-101

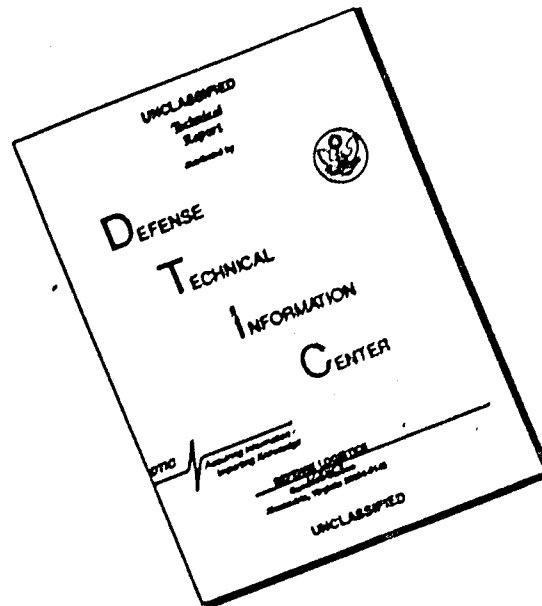
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AFML-TR-73-101

## GRAPHITE FIBER AND BORON NITRIDE FIBER FILLED POTTING COMPOUNDS

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FOREWORD

This report was prepared by the Composites and Fibrous Materials Branch, Nonmetallic Materials Division, Air Force Materials Laboratory. The work was conducted under Project No. 7340, "Nonmetallic and Composite Materials," Task No. 734004, "Structural Plastics and Composite Materials," with Mr. R. J. Dauksys serving as Project Engineer.

The author wishes to gratefully acknowledge R. Kuhbander for his contribution in the fabrication and testing of the potting compounds described in this report.

This report covers work from February 1972 to February 1973.

This technical report has been reviewed and is approved.



T. J. Reinhart, Jr., Acting Chief  
Composite and Fibrous Materials Branch  
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## ABSTRACT

Preliminary information and data are presented which show how the thermal, electrical, and mechanical properties of epoxy potting compounds may be controlled by the addition of discontinuous fibrous reinforcements of high modulus graphite and/or boron nitride (BN). The addition of 16 parts per hundred (pph) of nominal 3 mil long graphite fibers of  $83 \times 10^6$  psi modulus decreased the linear thermal coefficient of expansion ( $\alpha$ ) of an epoxy by 77% (from  $54.3 \times 10^{-6}$  in/in/ $^{\circ}\text{C}$  to  $12.3 \times 10^{-6}$  in/in/ $^{\circ}\text{C}$ ).

For the high modulus graphite fiber fiber modification compared to the unfilled epoxy potting compound, modulus was increased by a factor of approximately 2.8 ( $0.46 \times 10^6$  psi to  $1.28 \times 10^6$  psi) while strength was only marginally increased ( $15.7 \times 10^3$  psi to  $16.3 \times 10^3$  psi). BN fiber additions, up to 14 pph, had little effect on modulus or strength; however, for the 14 pph addition  $\alpha$  was reduced by approximately 40%.

The reduction of  $\alpha$  of an epoxy potting compound utilizing graphite and/or BN fibers is shown to be a function of modulus of fibers and negative  $\alpha$  of fibers along their length, as well as quantity of fibers used.

The addition of 14 pph boron nitride fibers only marginally reduced resistivity, i.e., to  $6.9 \times 10^{13}$ . Combinations of graphite and BN fibers provided epoxy potting compounds with properties intermediate compared to epoxy potting compounds with additions of graphite or BN fibers as sole modifier.

Density for the highest loaded epoxy (16 pph) containing the densest fiber modifier (graphite) with  $P=2.0$  g/cc) was only increased by approximately 5.5% over the unfilled epoxy potting compound. All potting formulations were of pourable consistency indicating good filling characteristics.



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## SECTION I

### INTRODUCTION

The materials development program described herein was initiated as a result of a request from the Space and Missiles Systems Organization (SAMSO) to act as consultant on a problem involving premature failure of epoxy potted sensors during fabrication of a classified reentry vehicle. In essence, approximate calculations indicated that during a bonding operation at elevated temperatures, the already epoxy potted sensors were exposed to thermally induced compressive forces far in excess of their rated capability. Possible solutions to the problem were:

- a. Utilization of a less pressure sensitive sensor;
- b. Utilization of a low temperature curing adhesive;
- c. Redesign of the reentry vehicle;
- d. Utilization of a lower-modulus/thermal-coefficient-of-expansion ( $E \alpha$ ) potting compound with similar handling characteristics as the one initially selected;
- e. Combinations of the above.

Item d is the subject of this report.

Epoxy potting compounds are widely used for a multitude of commercial, as well as aerospace applications. This report addresses potential problems associated with potting of delicate electrical, optical, and/or mechanical sensors. Through experiment, it is shown how an epoxy polymer may be modified with discontinuous high performance graphite and/or boron nitride fibers to achieve heretofore unrealized potting compound properties. Although the material developments discussed are oriented toward epoxy potting compounds, other classes of polymers may be modified in like manner to achieve similar improvements in mechanical properties or alteration in physical characteristics.

## SECTION II

## MATERIALS

## 1. Epoxy Potting Resin

A control epoxy potting compound was used throughout this program to study the effect of various fillers of different amounts on mechanical, physical, thermal, and electrical properties. The resin system (Reference 1) was designated Epon 815\*, a bisphenol-A epoxy which was modified with a mono-epoxy diluent to reduce viscosity. The curing agent was diethylene-triamine (DTA) added at 11 pph (Parts per hundred by weight). Properties of the cured, unmodified resin are shown in Table I.

## 2. Graphite Fibers

Discontinuous graphite fibers for modification of the epoxy potting compound were obtained from the Thornel\*\* Series of continuous yarns. Properties are shown in Table I. A carbon yarn designated VYB 105-1/5 of lower modulus than the Thornel yarns was also used. The Thornel 75 yarn had a measured average modulus of  $83 \times 10^6$  psi and  $330 \times 10^3$  psi tensile strength. Of note is the reported increase in negative  $\alpha$  up to the  $50 \times 10^6$  psi modulus fiber, and then the decrease in negative  $\alpha$  with  $75 \times 10^6$  psi filament (Reference 2). The slight difference in  $\alpha$  between Thornel 50 and Thornel 75 fibers is also shown to carry over to potting compounds of the same quantity fibers as described in subsequent sections of this report.

## 3. Boron Nitride (BN) Fibers

The BN fibers, supplied in mat form, were of an experimental lot obtained from Carborundum Corporation. Figure 1 shows the bulk mat in the "as-received" condition and Figure 2 shows fibers from the mat at higher magnification. Unlike the carbon base fibers, the BN has dielectric

---

\*Shell

\*\*Union Carbide

characteristics which impart attributes as an insulator rather than a conductor.

The reported coefficient of linear thermal expansion (Reference 2) was determined from X-ray analysis of single crystals. The actual value for the imperfect mat fibers is probably far less negative than indicated. Recent BF fiber developments indicate that much higher strength and modulus are possible than indicated in Table I. As the modulus increases and the fiber assumes a more ordered crystalline structure,  $\alpha$ , in the direction of the fiber would be expected to become more negative and approach the theoretical value shown in Table I.

## SECTION III

### MECHANICAL AND PHYSICAL PROPERTY EVALUATIONS

#### 1. Flexural Strength and Modulus

Flexural strength and modulus were obtained from the same specimens that were used for thermal expansion measurements. The tests were conducted using procedures of Federal Test Specification E-P 406b as a guide. The 3-point loading method was employed with a span-to-depth ratio of 16 to 1. Specimens were tested at a crosshead rate of 0.05 inches per minute.

#### 2. Density

Density was determined by water immersion.

#### 3. Resistivity

Resistivity measurements were made utilizing a Beckman Megohmmeter, Model L-8 following the procedures outlined in ASTM D-257-66. Samples were 3 inches in diameter and 1/8 inch thick.

#### 4. Viscosity

Viscosities were obtained using a Brookfield Viscometer, Model RVF, with a Number 6 spindle at 2, 4, 10, and 20 RPM's. ASTM D-1824-61T was used as a guide. The container used for the measurements was 4 inches in depth and 1.125 inches inside diameter. The potting compound was filled to 0.25 inches from the top and the spindle centered in the compound. Readings were taken at  $72 \pm 2^\circ\text{F}$ . As per ASTM D-1824-61T, comparison may be made to single reported values by using the viscosity determined at a spindle RPM of 20.

#### 5. Fiber Length Determinations

Fiber lengths were measured from 250X photographs of cured potting compounds. The average length of fibers, for all the potting compounds, was approximately 0.003 inch as indicated from the sample determinations

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shown below:

a. Fiber Length of 5 PPH Thornel 75

	A	B	C
Fiber Size (inches)	$<3.2 \times 10^{-3}$	$3.2-6.0 \times 10^{-3}$	$>6.0 \times 10^{-3}$
Average Length of Fiber in Categories A, B, +C	$1.65 \times 10^{-3}$	$4.59 \times 10^{-3}$	$8.29 \times 10^{-3}$
% of Fibers of Average Length in Categories A, B, +C	69.5	19.5	11.0

Average Fiber Length =  $2.95 \times 10^{-3}$  inches

b. Fiber Length of 5 PPH Thornel 50

	A	B	C
Fiber Size (inches)	$<3.2 \times 10^{-3}$	$3.2-6.0 \times 10^{-3}$	$>6.0 \times 10^{-3}$
Average Length of Fibers in Categories A, B, +C	$1.72 \times 10^{-3}$	$4.49 \times 10^{-3}$	$9.03 \times 10^{-3}$
% of Fibers of Average Length in Categories A, B, +C	80	10	10

Average Fiber Length =  $2.72 \times 10^{-3}$  inches

6. Coefficient of Linear Thermal Expansion

The test method and apparatus for determining the coefficient of linear thermal expansion of the materials in this investigation were similar to those described in ASTM D-696-70.



The specimen used was rectangular in cross section, having overall dimensions of 2.0 inches x 0.375 inch x 0.250 inch. The coefficient of expansion for the potting compounds was determined from  $-30 \pm 2^\circ\text{C}$  to  $+30 \pm 2^\circ\text{C}$ . A minimum of 2 tests was conducted for each value of  $\alpha$  shown in Table III.

## SECTION IV

### PREPARATION OF CURED POTTING COMPOUNDS

Several mixing methods were investigated. The results obtained for  $\alpha$  were not significantly changed regardless of the methods used. Methods were tried using hand mix, blenders, and vacuums, and combinations of each. The most satisfactory method was as follows:

The apparatus used for mixing the reinforcement and resin was a two-speed Waring blender with four cutting blades. The amount of resin (less curing agent) used must be sufficient to cover the cutting blades which in this case was 100 grams. After the resin is poured into the blender, the prepared chopped fibers were added. The continuous yarns, as well as BN fiber material were manually cut with a scissors to about one-half inch lengths and transferred to a container prior to loading the blender with resin. Best mixing was obtained by adding the chopped fibers in increments of 5 gph of resin.

The mixing time (10-25 sec) was generally controlled by the sound of the blender. Initial mixing caused cavitation and some fiber filler was deposited on the sides of the blender above the resin level. Mixing was stopped and the fibers were scraped from the sides and deposited in the resin. When the fibers are properly blended, the mixture no longer cavitates and the blender has a smooth mixing sound. Fibers of higher modulus appeared to require less mixing time.

During blending, the mixture increased in temperature due to heat transfer from the mixture motor assembly. Before the curing agent was added, it was important that the mixture be allowed to cool to room temperature. While the mixture was cooling, it was placed in a vacuum to remove entrapped air. After the mixture was cooled to room temperature the curing agent was added.

All compounds were cured at room temperature for a minimum of 16 hours prior to mechanical and physical testing.

## SECTION V

## CALCULATIONS

## 1. Thermally Induced Compression Stresses (Approximate)

The stresses within the potting compound and, in turn, on potted sensors were calculated for a 2-dimensionally restrained system, i.e., one face of potting compound unrestrained, and for a 3-dimensionally restrained system, i.e., the potting compound totally enclosed within a container. In both cases the container was assumed to have negligible expansion.

## a. The 2-Dimensionally Restrained Potting Compound

To approximate the stresses on an element in a potting compound the following Hooke's law relationships were used

$$e_x = \frac{1}{E} \sigma_x - \frac{\nu}{E} (\sigma_y + \sigma_z) + \alpha \Delta T \quad (1)$$

$$e_y = \frac{1}{E} \sigma_y - \frac{\nu}{E} (\sigma_x + \sigma_z) + \alpha \Delta T \quad (2)$$

$$e_z = \frac{1}{E} \sigma_z - \frac{\nu}{E} (\sigma_x + \sigma_y) + \alpha \Delta T \quad (3)$$

where  $e_x$ ,  $e_y$ ,  $e_z$  and  $\sigma_x$ ,  $\sigma_y$ ,  $\sigma_z$  represent the principal strains and stresses, respectively.  $E$  and  $\nu$  are the modulus and Poisson's ratio of the cured potting compound, respectively;  $\alpha$  is the coefficient of linear thermal expansion; and  $\Delta T$  is the change in temperature.

Assuming 2-dimensional restraint (Figure 3),  $z$ , being unrestricted, we have

$$e_x = e_y = 0 \quad (4)$$

$$e_x = \frac{1}{E} \sigma_x - \frac{\nu}{E} (\sigma_y + \sigma) + \alpha \Delta T = 0 \quad (5)$$

$$e_y = \frac{1}{E} \sigma_y - \frac{\nu}{E} (\sigma_x + \sigma_z) + \alpha \Delta T = 0 \quad (6)$$

$$\sigma_z = 0 \quad (7)$$

Solving simultaneously for  $\sigma_y$

$$\left( -\frac{\nu^2}{E} + \frac{1}{E} \right) \sigma_y = -(\nu+1) \alpha \Delta T \quad (8)$$

$$\sigma_y = \frac{-(\nu+1) \alpha \Delta T}{\left( -\frac{\nu^2}{E} + \frac{1}{E} \right)} \quad (9)$$

Using the following potting compound (unfilled) property values, we obtain

$$E = 0.46 \times 10^6 \text{ psi}$$

$$\nu = 0.35$$

$$\alpha = 54.3 \times 10^{-6} \text{ in/in/}^\circ\text{C}$$

For a  $1^\circ\text{C}$  rise in temperature ( $\Delta T$ ), the stress for the 2-dimensionally restrained compound is 38.5 psi (compression) or  $\sigma_x = \sigma_y = 38.5 \text{ psi}$

$$\sigma_z = 0$$

#### b. The 3-Dimensionally Restrained Potting Compound

If the z-plane restriction is considered, as shown in Figure 4, then

$$e_x = e_y = e_z = 0 \quad (10)$$

and Equations 1 - 3 may be consolidated, so that

$$\frac{1}{E} \sigma - \frac{\nu}{E} (2\sigma) + \alpha \Delta T = 0 \quad (11)$$

Rearranging, we can write

$$\sigma = - \frac{\alpha \Delta T}{\frac{1}{E} - \frac{2\nu}{E}} \quad (12)$$

and by using the properties for the unfilled epoxy potting compound listed in Table I above, a 1°C rise in temperature induces a compressive pressure of 82.3 psi or

$$\sigma_x = \sigma_y = \sigma_z = 82.3 \text{ psi}$$

(NOTE: No attempt was made to provide a rigorous analytical solution for the particular configuration investigated.) The subject potting container was of 6-4 titanium alloy ( $E = 16 \times 10^6$  psi,  $\alpha = 9.5 \times 10^{-6}$  in/in/°C), and it was assumed rigid, thus of no influence on the expansional characteristics of the potting compound. In fact, the expansional characteristics of the container would have a tendency to reduce compression stresses generated within the potting compound. The higher the  $\alpha$  of the container, the lower the compression stresses.

## 2. Flexural Modulus and Strength

Potting compound flexural strength was determined from

$$\sigma = \frac{3SP}{2WD^2}$$

where

$\sigma$  = flexural strength (pounds/inches<sup>2</sup>)

$S$  = span (inches)

$P$  = load (pounds)

$W$  = width (inches)

$D$  = depth (inches)

Modulus was calculated from

$$E = \frac{PS^3}{4WD^3}$$

where the symbols are as defined for the preceding flexural strength equation.

3. Factor Increase in Weight of Various Powder Fillers Over Weight of Thorne1 50 Fibers to Obtain  $\alpha$  of  $13.4 \times 10^{-6}$  In/In/°C

The results shown in Column A of Table IV were obtained by dividing the percent total weight of filler (extrapolation of curves of Figure 6) by the total weight percent (11.2%, 14 pph) Thorne1 50 to obtain a value of  $13.4 \times 10^{-6}$  in/in/°C.

4. Factor Increase in Density of Various Potting Compounds Over Density of Thorne1 50 Potting Compound to Obtain  $\alpha$  of  $13.4 \times 10^{-6}$  In/In/°C

The results shown in Column B of Table IV were obtained by calculating the densities of the various modified potting compounds and then dividing each calculated density by the measured density of the 11.2% by weight Thorne1 50 fiber filled potting compound. The Rule-of-Mixtures was used to calculate densities; for example

$$\rho_{pc} = \rho_f W_f + \rho_r W_r$$

where

$\rho_{pc}$  = density of potting compound

$\rho_f$  = density of fiber

$\rho_r$  = density of resin

$W_f$  = percent by weight fiber

$W_r$  = percent by weight resin  
(including curing agent)

## SECTION VI

## DISCUSSION

Fillers are used extensively to modify epoxies to obtain desired end-properties. Modification of potting compounds to reduce  $\alpha$  and potential thermally induced stresses on delicate embedded components, has been accomplished by compounding the resin system with fillers such as aluminum oxide, aluminum powders, mica, talc, silica, lithium aluminum silicate, calcium carbonate, and others. Figure 5 shows the reduction of  $\alpha$  versus amount of a particular filler. It is apparent that, to obtain significant reductions in  $\alpha$ , filler in very large quantities is required. Figure 6 consists of another series of curves obtained from a different source (Reference 1) which show that the compound must be heavily filled to obtain appreciable decreases in  $\alpha$ . Because of this fact, the improvement (reduction) in  $\alpha$  is countered by undesirable changes in properties such as increased viscosity, density, and modulus. The latter influences the degree of thermally induced stresses.

The maximum amount of fibers compounded was limited to 16 pph (12.6% by weight of total) to maintain a pourable consistency. Table II shows viscosities in centipoises for the uncured potting compounds. The value at a spindle RPM of 20 is customarily reported. For the potting compound application which led to this study, 20,000 centipoises was a requirement, although a higher value (other requirements being satisfied) could be tolerated.

Figure 7 shows  $\alpha$  versus quantity of fibers for the systems investigated. Increasing amounts (to 14 pph) of negative  $\alpha$  BN fibers indicates a decreasing  $\alpha$  of the potting compound. The same is true for the Thornel fiber modifications, where the greater negative  $\alpha$  fibers impart the lowest  $\alpha$  potting compounds. The  $\alpha$ 's of the Thornel 50 and Thornel 75 fibers (Table I) are numerically very close. This similarity is also reflected in potting compounds containing the different fibers, but comparable weight fractions. Only one series of thermal expansion measurements was conducted for the low modulus ( $6 \times 10^6$  psi) carbon

fiber modified epoxy potting and is shown in Figure 7 by the solid triangle.

The effect on potting compound  $\alpha$ , using combinations of Thornel 75 fibers and BN fibers, is shown in Figure 8. It appears that a linear relationship exists between the limits of 0 and 14 pph fibers and that mixtures of these fibers within those limits will provide intermediate  $\alpha$ 's.

Calculations were made (Section V) to approximate the thermally induced compression stresses generated on an encapsulated component for a  $1^\circ\text{C}$  rise in temperature. Results are depicted in Table III for the various fiber-modified potting compounds. These same results were used to plot the compression stresses versus temperature rise from room temperature ( $22^\circ\text{C}$ ) curves (Figures 9-13) for the different systems investigated. Both 2- and 3-Dimensionally restrained compounds are shown. The figures graphically illustrate the importance of temperature control and design of the potting container (restraint effects) to maintain stresses at the lowest possible level.

As indicated by the curves, one approach to reduce stresses would be to provide a design that does not warrant intimate contact of the potting compound with all sides of the container. As shown in the curves, the 2-dimensionally restrained compound allows stress relief, whereas the 3-dimensionally restrained system does not. When permissible, stresses may be further reduced by closely matching the  $\alpha$  of the potting compound to that of the container.

The magnitude of stresses is not solely attributed to  $\alpha$ , but is actually the product of modulus and  $\alpha$  or  $E\alpha$ . This is clearly indicated when we compare (Table III) the stresses per centigrade degree rise in temperature for the 5 pph Thornel 75 compound to the 16 pph Thornel 75 compound. Even though the 16 pph compound is one half the  $\alpha$  of the 5 pph compound, the significantly higher modulus of the 75 pph modification, due to higher value fraction fibers, results in higher stresses.



Alternately, The  $\alpha$  influence on thermally induced stresses are shown when we compare potting compounds of equivalent weight fractions fiber reinforcement, but different modulus fiber. For instance, 16 pph of the lower modulus Thorne! 50 results in lower thermal stresses than the epoxy formulated with 16 pph of the higher modulus Thorne! 75 (recall that  $\alpha$  of the Thorne! 50 fiber is approximately the same as  $\alpha$  of the Thorne! 75 fiber).

It is cautioned, that the calculated compression stresses for both a 3-dimensionally restrained (totally enclosed) and 2-dimensionally restrained (no restriction on one face) potting compounds are only approximate, because we assumed that the container material was completely rigid and nonexpanding. Depending on the  $\alpha E$ , the geometry, and thickness of the container material, stresses may be further reduced. However, the results on a qualitative basis, are quite informative for rating potting compounds and indicating the potential severity of thermally induced stresses on sensitive potted elements.

The slight discrepancies in modulus values at the highest fiber loadings (14 and 16 pph) are attributed to experimental error in preparing the compounds of slight volume fraction difference. Typically, the modulus would be expected to increase for the higher fiber loading. Another possible source of error is that the specimens did not all have the same length of cure. This would also affect the modulus of the potting compounds.

Referring to Table III, it is seen that a wide range of  $\alpha$ 's are possible, and depending on requirements, resistivity may also be controlled. Because of the dielectric properties, BN fiber potting compound modifications offer not only reduction of  $\alpha$  and thermal compression stresses, but also good dielectric characteristics such as indicated by the negligible loss of resistivity for the 14 pph addition. Although the hexagonal crystal structure of BN (Reference 3) is a close analog of the graphite structure, and many properties are similar (theoretical mechanical properties, high temperature resistance, and

lubricating qualities), other properties are radically different. For instance, BN is white and has exceptionally good insulating properties while graphite is black and behaves as a conductor. At room temperature the specific resistance of BN has been reported in excess of  $10^{12}$  ohms. Anticipated developments in BN fiber technology will provide fibers with higher strength, modulus, and lower thermal coefficient of expansion without affecting its excellent dielectric properties. Thus, for an application requiring high insulating qualities, as might be expected in potting unprotected electrical components, a BN fiber formulation may be in order.

As mentioned earlier, another advantage of compounding with these discontinuous high performance fibers is that the weight per unit volume is changed little over the control unmodified potting compound because (a) such a small quantity of fibers is necessary to achieve desired properties, and (b) the fibers are themselves of low density. Table IV lists typical fillers which have been used to modify potting compounds and notes those that are capable of providing  $\alpha$ 's as low as that obtained ( $13.4 \times 10^{-6}$  in/in/ $^{\circ}\text{C}$ ) for the 11.2% by weight Thorne 50 fiber modification. The total weight percent modifier indicated to reduce  $\alpha$  was determined by extrapolating the curves of Figure 6 to  $13.4 \times 10^{-6}$  in/in/ $^{\circ}\text{C}$ . The differences between the  $\alpha$  and density of the Epon 828 system (Figure 6) and the control Epon 815 system used in this study were considered negligible. It is assumed that the high weight percent fillers shown in Figure 6 would be difficult to compound and be of extremely high viscosity.

The A\* column of Table IV indicates, for the fillers listed, that to achieve an  $\alpha$  of  $13.4 \times 10^{-6}$  in/in/ $^{\circ}\text{C}$ , at least seven times as much filler as the Thorne 50 addition would be required. Further, on a weight-per-unit-volume basis we see from Column B\*, Table IV, that the additional quantity of filler required with the corresponding higher density than graphite (or BN) would yield compounds significantly denser than the graphite fiber filler modification.

The graphite fibers, although well established as high performance fibrous reinforcements, have yet to develop a sizable market to reduce current prices of about \$50 per pound and up, depending on modulus of yarn and construction. It is anticipated that further developments in precursor technology and production processes could reduce the cost to \$5-10 per pound for continuous fibers. It may be possible to produce or obtain short fibers of the type necessary to obtain low  $\alpha$  potting compounds for less cost.

The BN fibers have not been developed to the degree that graphite fibers have, i.e., controllable modulus in excess of  $80 \times 10^6$  psi. Future advancements in this area promise to provide fibers with significantly higher modulus and strength properties and correspondingly more negative values of  $\alpha$  than the fibers used in this preliminary study. Projected economics indicate that it is feasible to produce continuous BN fibers of high modulus and strength for less than \$5 per pound.

The current prices of graphite and BN fibers are not competitive with the fillers listed in Table IV, which sell for about 8-12 cents per pound in large lots. However, in some instances, due to the quantity of filler required, it may be impractical to utilize the low cost fillers because of processing restraints. Then too, the large filler addition (depending on type used) would increase modulus of potting compound, lessening, in turn, resistance to thermally induced stresses. Use of graphite and/or BN fibers as fillers certainly should be considered when combinations of attributes such as low density, viscosity, modulus, low quantity of fibers, control of thermal stresses, control of resistivity, and facile processing are desirable. Close scrutiny must be given to the seemingly obvious advantage of low cost of other fillers as compared to the resulting system performance.

Critical applications, aerospace or other, where reliability, survivability, and low maintainability are of paramount concern, dictate consideration of graphite and or BN fiber filler potting compounds.

## SECTION VII

### CONCLUSIONS

Preliminary experiments and tests have shown that it is feasible to incorporate small quantities (16 pph or less) of short fibers of graphite and/or boron nitride in an epoxy polymer to:

- a. Reduce  $\alpha$  significantly from  $54.3 \times 10^{-6}$  in/in/°C for the cured unmodified epoxy to  $12.3 \times 10^{-6}$  in/in/°C for a 16 pph Thornel 75 formulated compound.
- b. Considerably reduce thermally induced compression stresses on potted sensors or elements.
- c. Control  $\alpha$  over a wide range while maintaining low cured potting compound density, as well as pourable viscosity.
- d. Maintain high levels of potting compound resistivity by utilization of BN fibers as sole modifier.
- e. Adjust mechanical, physical, and thermal properties of potting compounds by using mixtures of graphite and BN fibers.

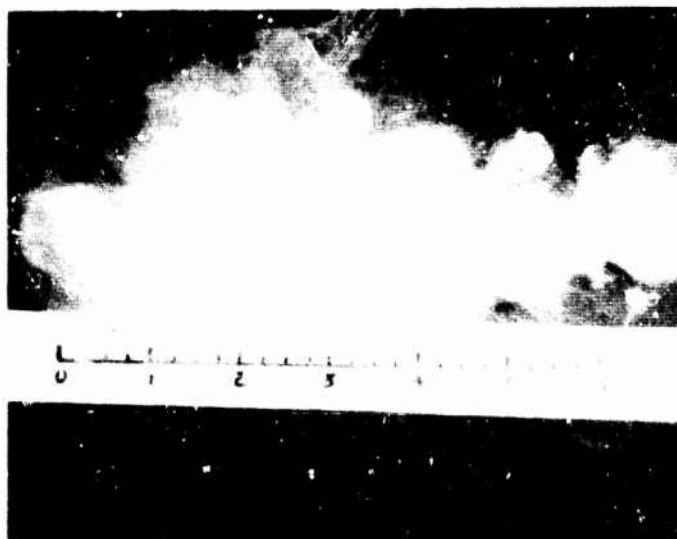


Figure 1. As-Received Boron Nitride Fiber Mat

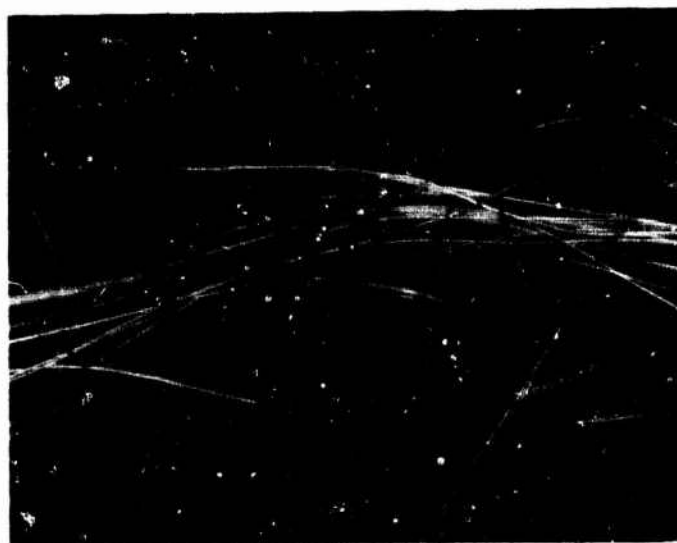


Figure 2. Higher Magnification of Boron Nitride Fibers from Mat

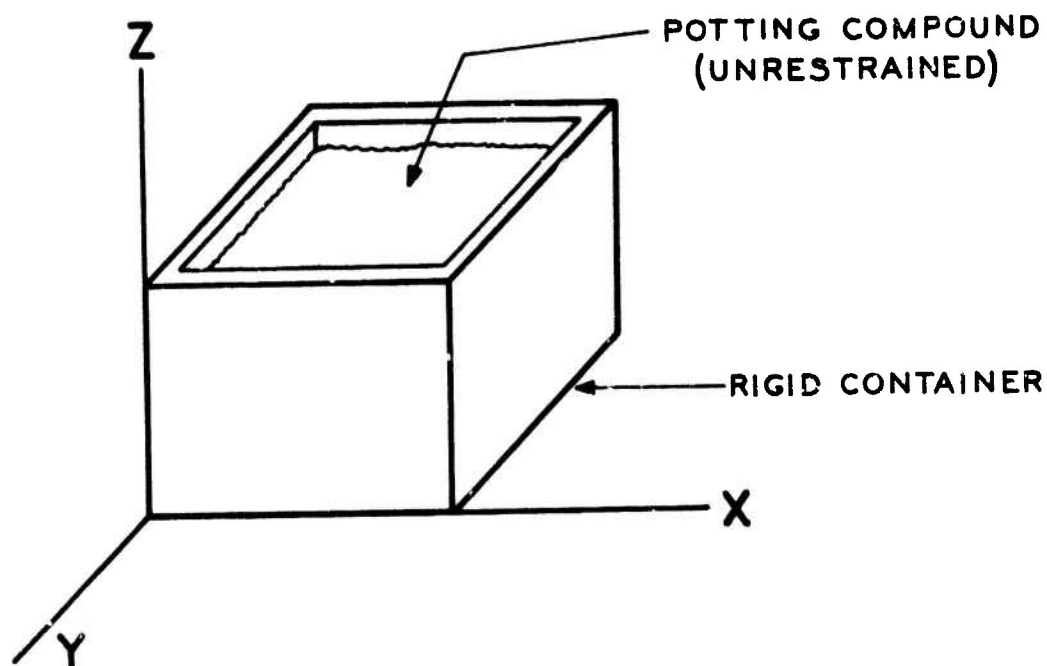


Figure 3. 2-Dimensionally Restrained Potting Compound

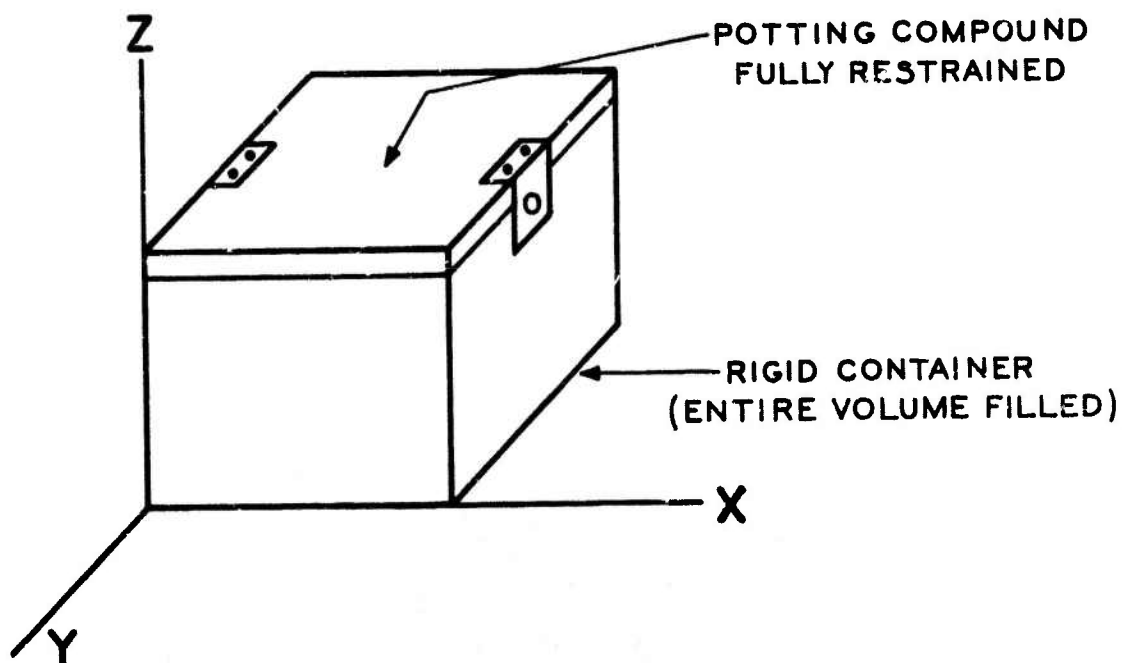


Figure 4. 3-Dimensionally Restrained Potting Compound

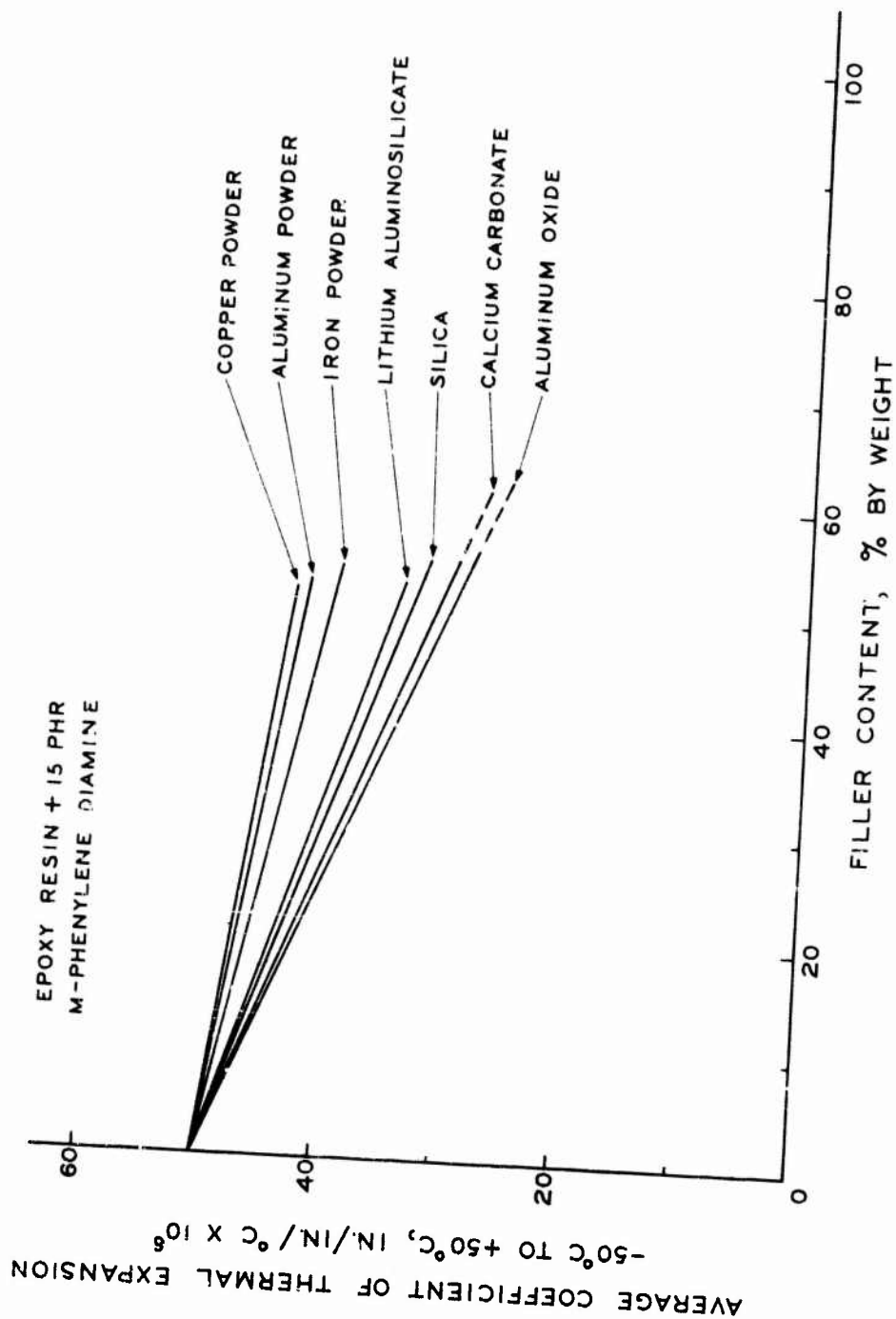


Figure 5. Effect of Fillers on Coefficient of Linear Thermal Expansion  
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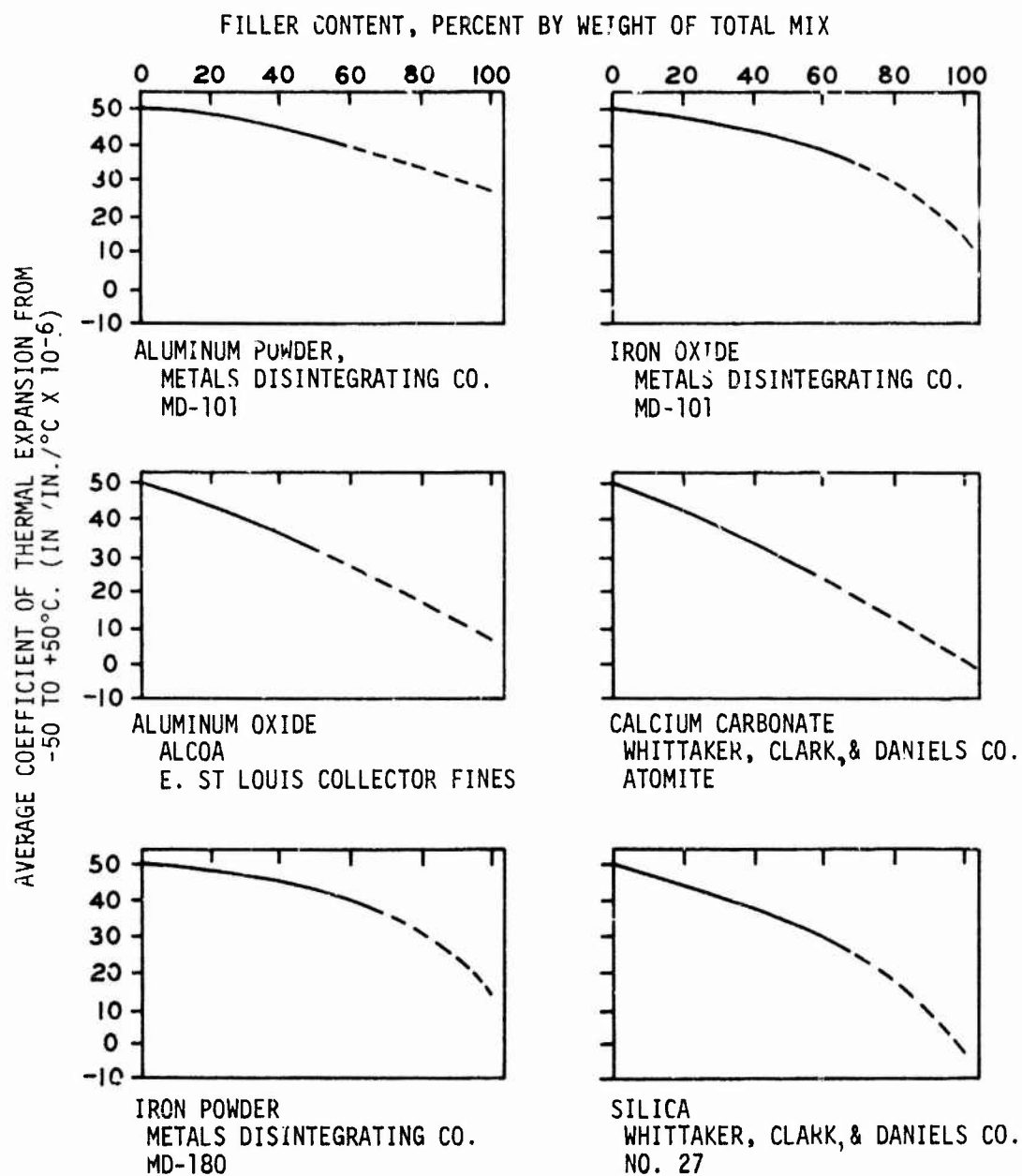


Figure 6. Shell Data. Effect of Fillers on Coefficient of Linear Thermal Expansion



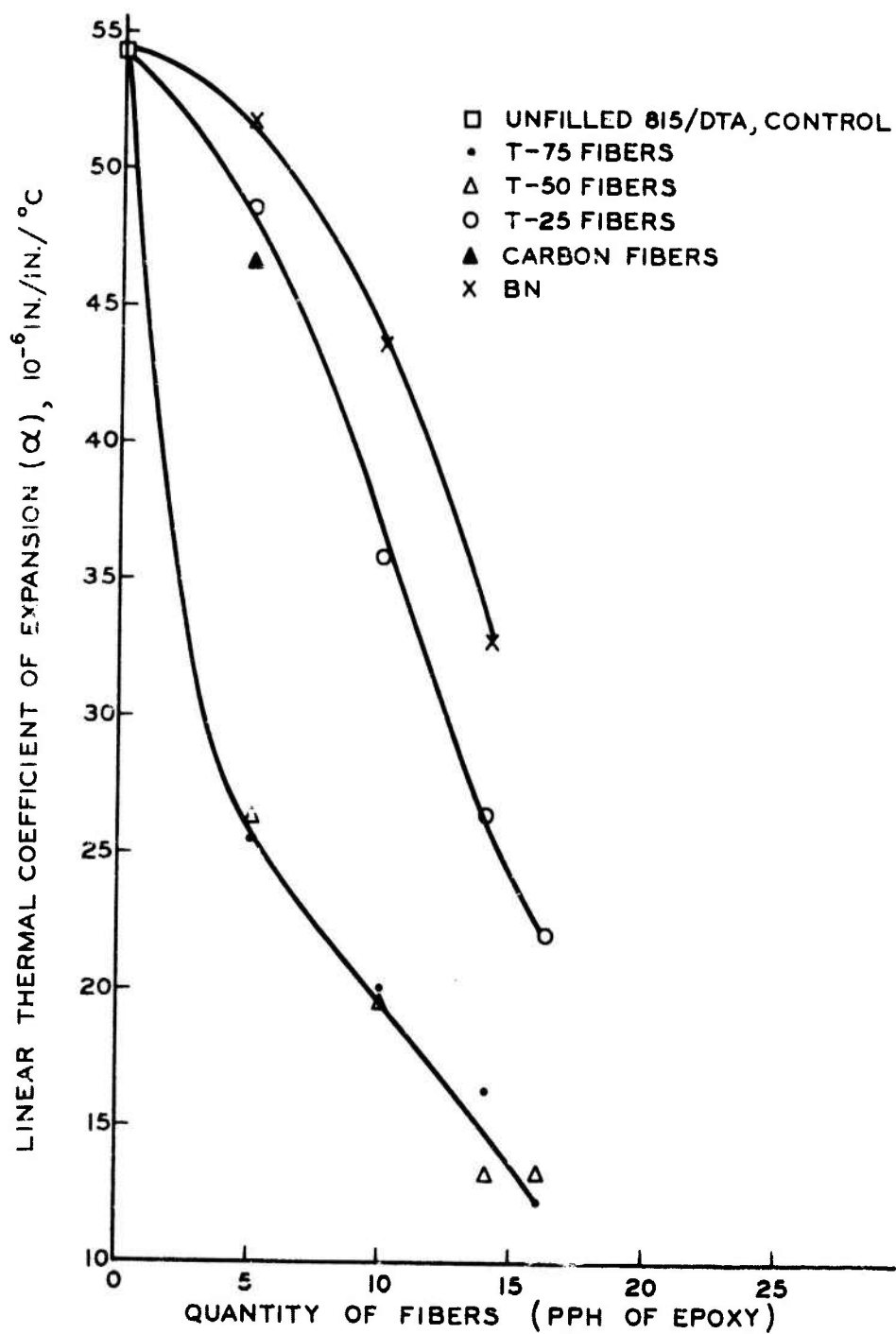


Figure 7. Effect of Graphite Fiber and BN Fiber Fillers on Coefficient of Linear Thermal Expansion of Epon 815/DTA

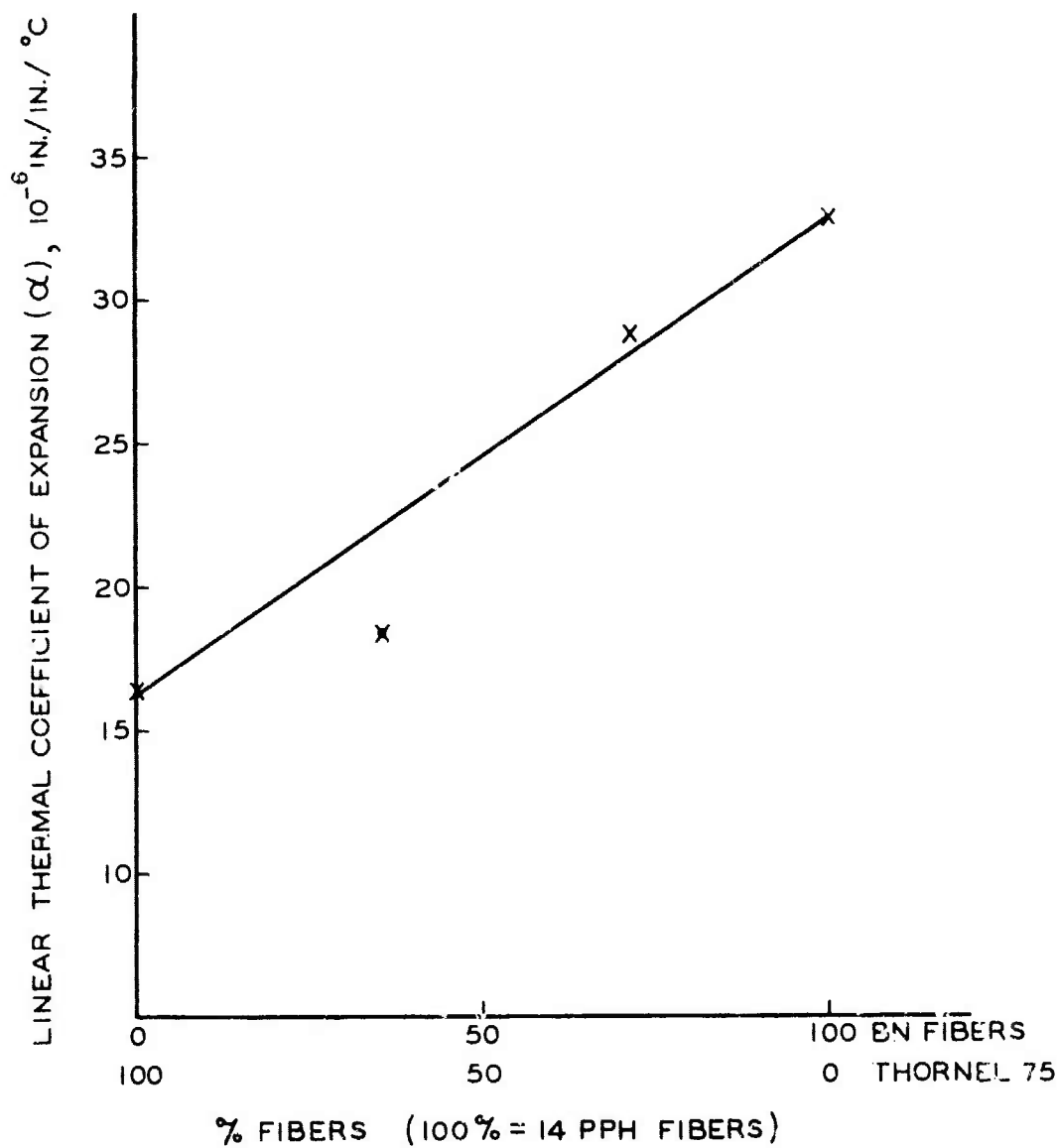


Figure 9. Effect of Mixtures of Graphite and BN Fiber Fillers on Coefficient of Linear Thermal Expansion of 815/CTA

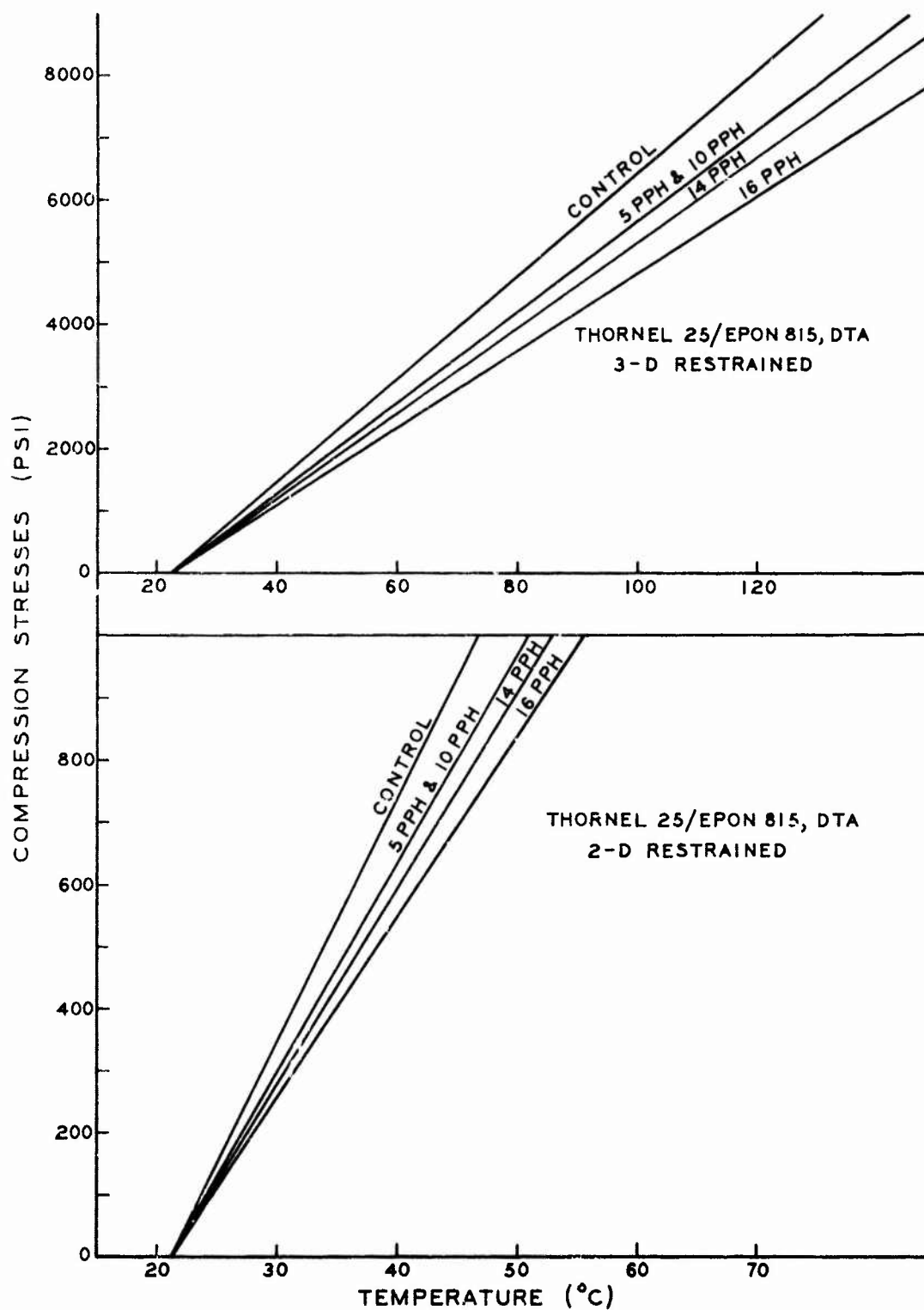


Figure 9. Thermally Induced Compression Stresses - Thornel 25 Fiber Filled/Epon 815, DTA-2-D and 3-D Restraints

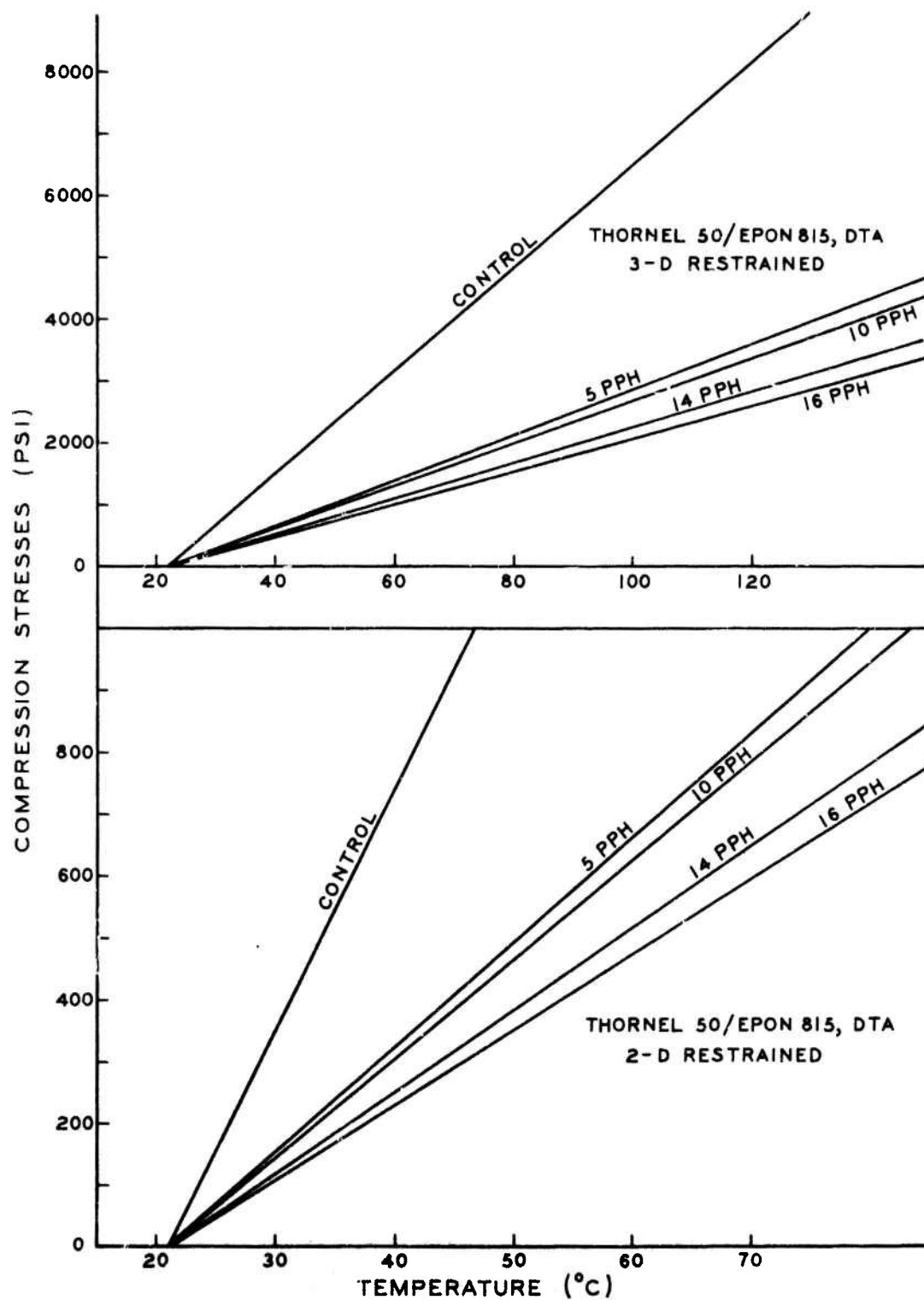


Figure 10. Thermally Induced Compression Stresses - Thornel 50 Fiber Filled/Epon 815, DTA-2-D and 3-D Restraints

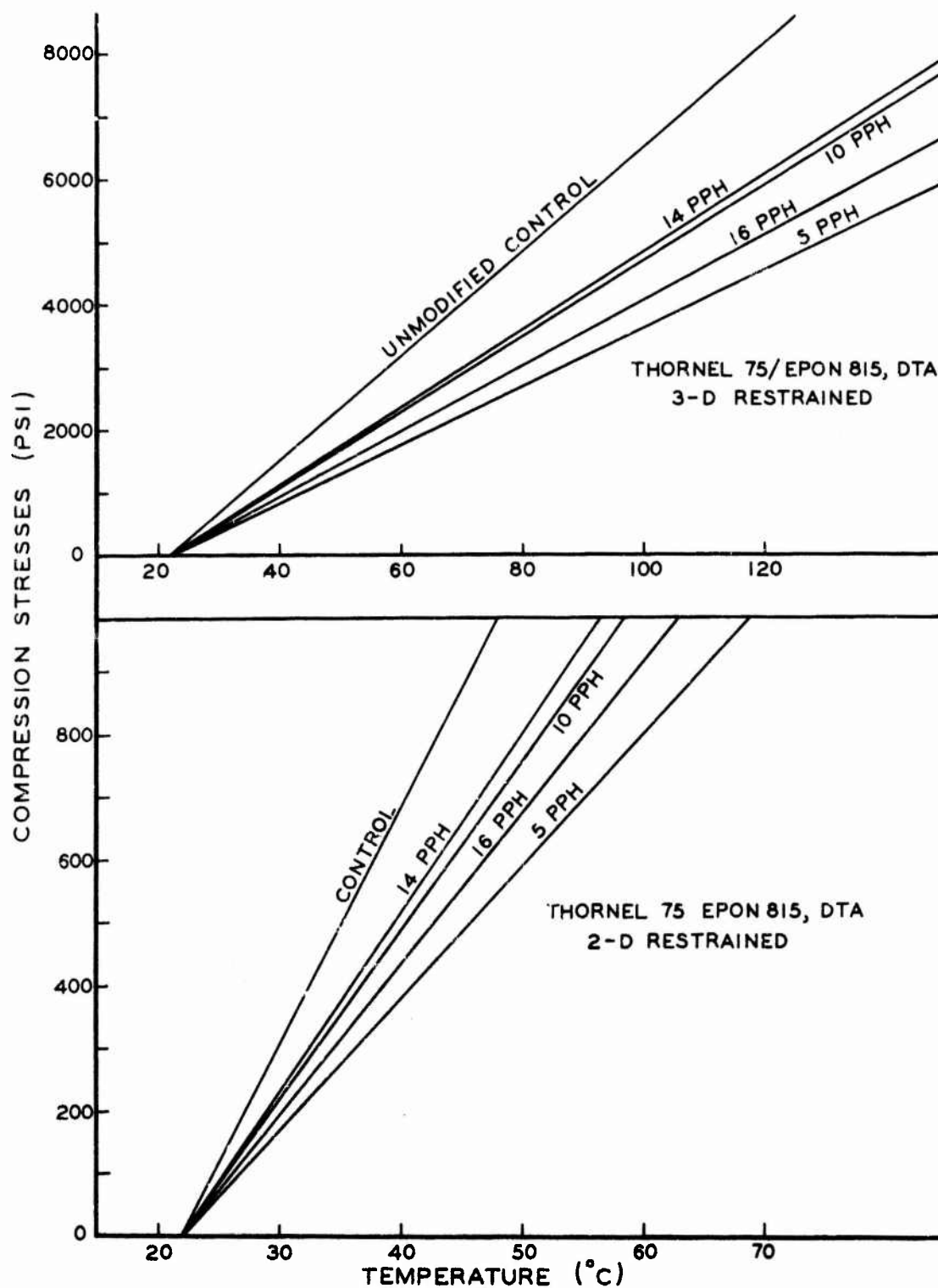


Figure 11. Thermally Induced Compression Stresses - Thornel 75 Fiber Filled/Epon 815, DTA-2-D and 3-D Restraints

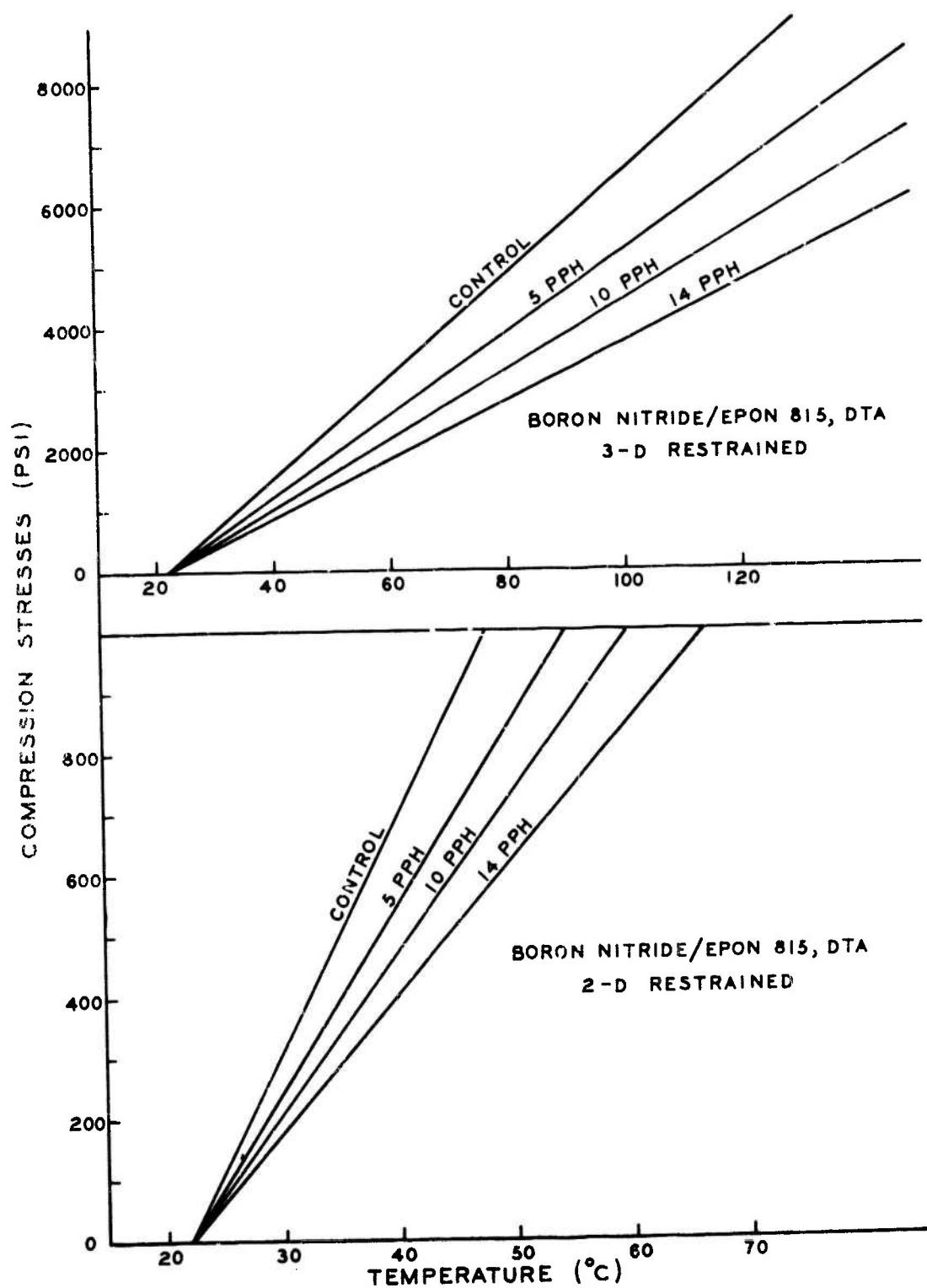


Figure 12. Thermally Induced Compression Stresses - Boron Nitride Fiber Filled/Epon 815, DTA-2-D and 3-D Restraints

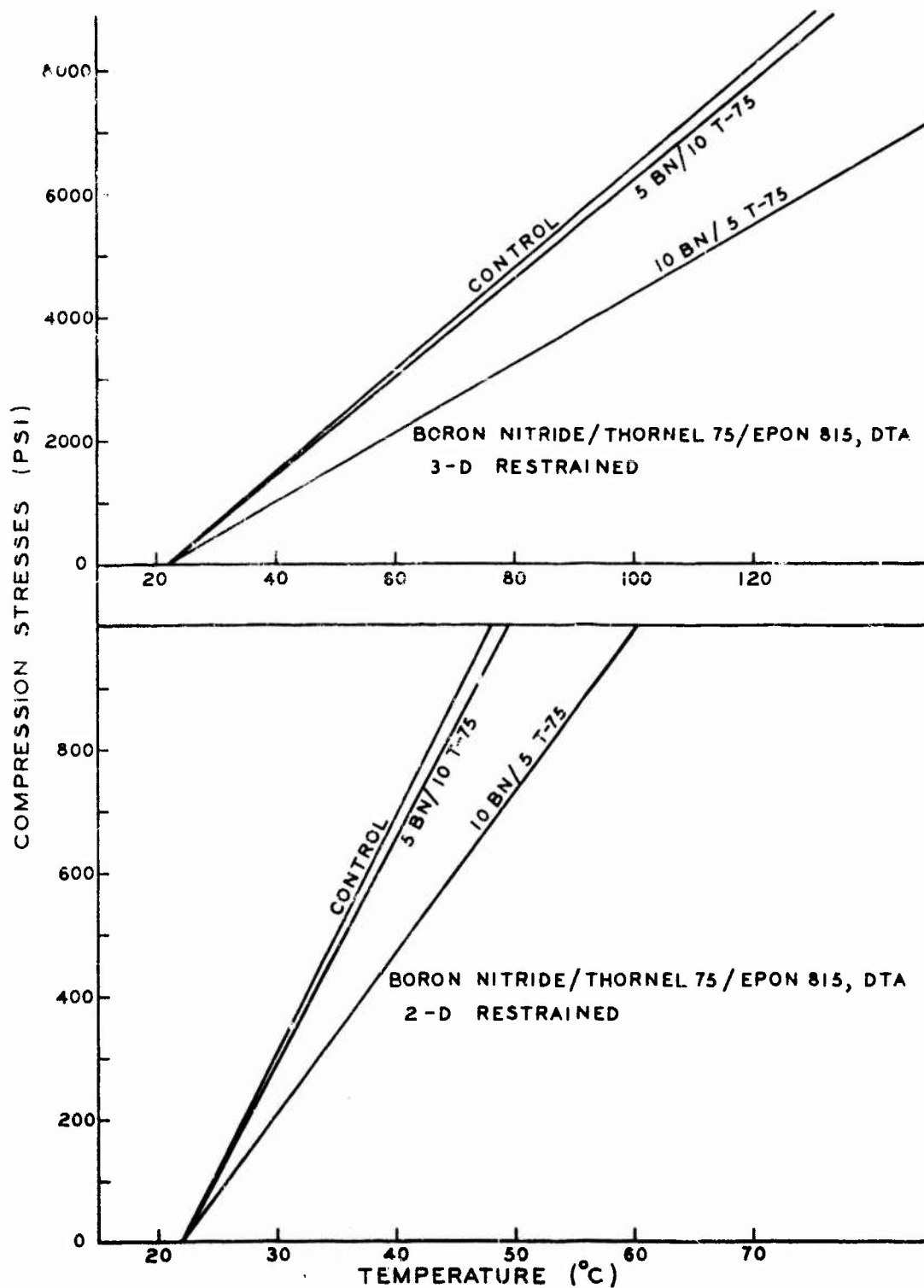


Figure 13. Thermally Induced Compression Stresses - Boron Nitride and Graphite Fiber Filled/Epon 815, DTA-2-D and 3-D Restraints

TABLE I  
PROPERTIES OF POTTING COMPOUND CONSTITUENTS

Property	CONSTITUENT MATERIALS					
	Carbon VYB 105-1/5	Thornel 25	Thornel 50	Thornel 75	Boron Nitride	Epon 815/DTA
Tensile Modulus $10^6$ psi	6	25	50	75	4-6	.46
Tensile Strength $10^3$ psi	180	180	320	375	50-100	15.7
Density g/cc	1.32	1.42	1.68	1.85	1.8-2.0	1.16
Coef. of Linear* Thermal Expansion $10^{-6}$ in/in/°C	---	-.445	-.800	-.733	$-2.9 \times 10^{-5}$	54.3
Yarn Construction plys	5	2	2	2	Supplied in mat form	----
filaments/ply	480	720	720	720		----
Twist/inch	1.1	1.5	1.5	1.5		----

\*Graphite  $\alpha$  values obtained from Reference 2.

Boron Nitride  $\alpha$  value from single crystal data. See Reference 2.



TABLE II  
 VISCOSITY IN CENTIPOISES FOR UNCURED  
 POTTING COMPOUNDS

Fiber	PPH Fiber	SPINDLE RPM			
		2	4	10	20
Control	0	-----	-----	-----	600
Carbon	5	21,000	18,250	11,800	7,300
BN	5	4,000	4,000	2,900	2,600
BN	10	6,000	4,500	3,500	2,850
Thorne 1 50	5	2,000	2,000	1,500	1,500
Thorne 1 50	10	51,500	26,250	12,400	8,000
Thorne 1 50	14	52,500	27,500	18,100	12,950
Thorne 1 50	16	56,500	34,500	18,500	15,050
Thorne 1 75	5	12,000	6,250	4,200	2,650
Thorne 1 75	10	15,000	10,000	6,400	4,450
Thorne 1 75	14	50,500	32,250	20,400	13,700
Thorne 1 75	16	66,500	44,750	31,400	22,300
BN/Thorne 1 75	5/10	57,500	36,500	25,000	18,300
BN/Thorne 1 75	10/5	56,500	29,000	19,000	14,800

TABLE III  
PROPERTIES OF DISCONTINUOUS GRAPHITE FIBER  
AND OR BORON NITRIDE FIBER MODIFIED EPOXY POTTING COMPOUNDS

Modification	PPH	Density g/cc	$\alpha$ $10^{-6}$ in/in/ $^{\circ}$ C	Modulus (E)* $10^6$ psi	Strength* $10^3$ psi	Compression Stresses, psi/ $^{\circ}$ C 2-0	Compression Stresses, psi/ $^{\circ}$ C 3-0	Resistivity ohm/cm
Control (Epon 815/DTA)	0	1.16	54.3	.46	15.7	38.5	82.3	$8.7 \times 10^{14}$
Carbor	5	1.16	46.7	.36	13.0	25.9	56.0	-
ThorneI 25	5	1.17	48.6	.45	8.5	33.6	73.0	-
ThorneI 25	10	1.01	35.8	.61	11.3	33.7	72.9	-
ThorneI 25	14	1.44	26.4	.78	12.6	31.7	68.6	-
ThorneI 25	16	1.19	26.9	.69	13.7	28.6	61.9	-
ThorneI 50	5	1.19	26.3	.42	12.9	17.0	36.9	$6.7 \times 10^8$
ThorneI 50	10	1.19	19.7	.53	11.4	16.0	34.8	$11.4 \times 10^9$
ThorneI 50	14	1.18	13.4	.65	13.8	13.4	29.0	$18.9 \times 10^9$
ThorneI 50	16	1.18	13.4	.60	12.6	12.4	26.8	$7.6 \times 10^8$
ThorneI 75	5	1.19	25.5	.54	12.1	21.1	46.4	$1.14 \times 10^{10}$
ThorneI 75	10	1.22	19.9	.90	12.9	27.8	60.2	$1.14 \times 10^9$
ThorneI 75	14	-----	16.4	1.13	15.3	28.5	62.0	$1.18 \times 10^9$
ThorneI 75	16	1.23	12.3	1.28	16.3	24.2	52.3	$4.9 \times 10^6$
BN	5	1.18	51.7	.39	13.0	30.9	67.0	$4.93 \times 10^{14}$
BN	10	1.20	43.7	.39	8.9	26.2	56.8	$3.00 \times 10^{15}$
BN	14	1.21	32.3	.44	13.1	22.2	48.0	$6.9 \times 10^{13}$
BN/ThorneI 75	5/10	1.20	18.4	.92	19.0	26.1	56.2	$9 \times 10^{108}$
BN/ThorneI 75	10/5	1.19	28.8	.84	13.6	37.2	80.7	$1.06 \times 10^7$
*Flexural modulus and strength								

TABLE IV  
QUANTITY-DENSITY COMPARISONS OF POTTING COMPOUNDS  
TO OBTAIN  $\alpha$  of  $13.4 \times 10^{-6}$  in/in/ $^{\circ}\text{C}$ \*

Filler	Density of Filler g/cc	$\alpha$ of Filler $10^{-6}$ in/in/ $^{\circ}\text{C}$	% (Total Weight) Filler to Obtain $13.4 \times 10^{-6}$ in/in/ $^{\circ}\text{C}$	A*	B*
Aluminum Powder	2.70	24	Not Possible	Not Applicable	Not Applicable
Aluminum Oxide Powder	3.97	7	90	8.03	3.12
Calcium Carbonate	2.93	0.7	80	7.14	2.18
Copper Powder	8.92	17	Not Possible	Not Applicable	Not Applicable
Iron Powder	7.86	11	95	8.49	6.37
Lithium Aluminum Silicate	2.38	0.3	80	7.14	1.81
Mica	3.40	22	Not Possible	Not Applicable	Not Applicable
Thornel 50 Graphite Fibers	1.68	-0.800	11.2	Not Applicable	Not Applicable

\* $13.4 \times 10^{-6}$  in/in/ $^{\circ}\text{C}$ ---Value of  $\alpha$  obtained with 11.2% (of total weight) Thornel 50.  
A--Factor increase in filler (over 11.2% by weight Thornel 50) to obtain  $13.4 \times 10^{-6}$  in/in/ $^{\circ}\text{C}$ .  
B--Factor increase in weight/volume of pottling compound to obtain  $13.4 \times 10^{-6}$  in/in/ $^{\circ}\text{C}$ .

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1. "Epon Resins for Castings, "Shell Chemical Company Catalogue, 1967.
2. Goodyear Aerospace Corporation Report GER-14781 S/3, May 1970.
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13. ABSTRACT <p>Preliminary information and data are presented which show how the thermal, electrical, and mechanical properties of epoxy potting compounds may be controlled by the addition of discontinuous fibrous reinforcements of high modulus graphite and/or boron nitride (BN). The addition of 16 parts per hundred (pph) of nominal 3 mil long graphite fibers of <math>83 \times 10^6</math> psi modulus decreased the linear thermal coefficient of expansion (<math>\alpha</math>) of an epoxy by 77% (from <math>54.3 \times 10^{-6}</math> in/in/°C to <math>12.3 \times 10^{-6}</math> in/in/°C).</p> <p>For the high modulus graphite fiber fiber modification compared to the unfilled epoxy potting compound, modulus was increased by a factor of approximately 2.8 (<math>0.46 \times 10^6</math> psi to <math>1.28 \times 10^6</math> psi) while strength was only marginally increased (<math>15.7 \times 10^3</math> psi to <math>16.3 \times 10^3</math> psi). BN fiber additions, up to 14 pph, had little effect on modulus or strength; however, for the 14 pph addition <math>\alpha</math> was reduced by approximately 40%.</p> <p>The reduction of <math>\alpha</math> of an epoxy potting compound utilizing graphite and/or BN fibers is shown to be a function of modulus of fibers and negative <math>\alpha</math> of fibers along their length, as well as quantity of fibers used.</p> <p>The addition of 14 pph boron nitride fibers only marginally reduced resistivity, i.e., to <math>6.9 \times 10^{13}</math>. Combinations of graphite and BN fibers provided epoxy potting compounds with properties intermediate compared to epoxy potting compounds with additions of graphite or BN fibers as sole modifier.</p>		

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13 ABSTRACT

Continued

Density for the highest loaded epoxy (16 pph) containing the densest fiber modifier (graphite) with  $P=2.0$  g/cc) was only increased by approximately 5.5% over the unfilled epoxy potting compound. All potting formulations were of pourable consistency indicating good filling characteristics.

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KEY WORDS

LINK A

LINK B

LINK C

ROLE

WT

ROLE

WT

ROLE

WT

Potting compounds  
Encapsulating  
Thermal expansion  
Thermally induced stresses  
Epoxy  
Boron nitride fibers  
Graphite fibers